

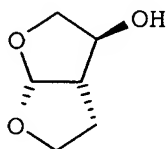
WHAT IS CLAIMED IS:

1. A method of preparing (3R,3aS,6aR)-3-hydroxyhexahydrofuro[2,3-b]furan comprising:

(a) optionally reacting (5S)-hydroxymethyl-5H-furan-2-one with a compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one to provide a protected furan-2-one;

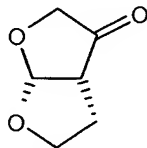
(b) subjecting (5S)-hydroxymethyl-5H-furan-2-one or the protected furan-2-one of optional step (a) to a photochemical reaction in the presence of 1,3-dioxolane to provide a 1,3-dioxolan-substituted furan-2-one;

(c) reducing the 1,3-dioxolane-substituted furan-2-one of step (b) to provide a reduced product, then hydrolyzing the reduced product to provide a product having a structure



;

(d) oxidizing the product of step (c) to provide a product having a structure



; and

(e) reducing the product of step (d) to provide (3R,3aS,6aR)-3-hydroxyhexahydrofuro[2,3-b]-furan.

2. The method of claim 1 wherein the (3R,3aS,6aR)-3-hydroxyhexahydrofuro[2,3-b]furan is prepared in at least 90% diastereomeric ratio.

3. The method of claim 1 wherein the (3R,3aS,6aR)-3-hydroxyhexahydrofuro[2,3-b]furan is prepared in at least 95% diastereomeric ratio.

4. The method of claim 1 wherein the compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one is selected from the group consisting of an acid chloride, a trialkylsilyl chloride, and a pyran.

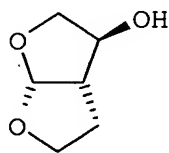
5. The method of claim 1 wherein the compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one is selected from the group consisting of tert-butyldimethylsilyl chloride, trimethylsilyl chloride, acetyl chloride, pivaloyl chloride, benzoyl chloride, methoxymethanol, benzyl alcohol, and dihydropyran.

6. A method of preparing (3R,3aS,6aR)-3-hydroxyhexahydrofuro[2,3-b]furan comprising:

(a) optionally reacting (5S)-hydroxymethyl-5H-furan-2-one with a compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one to provide a protected furan-2-one;

(b) subjecting (5S)-hydroxymethyl-5H-furan-2-one or the protected furan-2-one of optional step (a) to a photochemical reaction in the presence of 1,3-dioxolane to provide a 1,3-dioxolan-substituted furan-2-one;

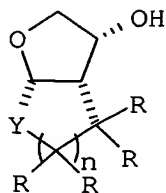
(c) reducing the 1,3-dioxolane-substituted furan-2-one of step (b) to provide a reduced product, then hydrolyzing the reduced product to provide a product having a structure



(d) subjecting the product of step (c) to a Mitsunobu inversion utilizing triphenylphosphine, *para*-nitrobenzoic acid, and diisopropylazodicarboxylate; and

(e) saponifying the product of step (d) to provide (3R,3aS,6aR)-3-hydroxyhexahydrofuro[2,3-b]furan.

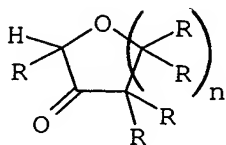
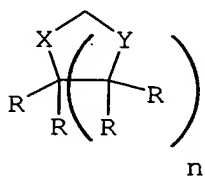
7. A method of preparing a compound having a structure



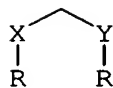
comprising:

(a) optionally reacting (5S)-hydroxymethyl-5H-furan-2-one with a compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one to provide a protected furan-2-one;

(b) subjecting (5S)-hydroxymethyl-5H-furan-2-one or the protected furan-2-one of optional step (a) to a photochemical reaction in the presence of a compound having a structural formula

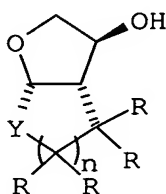


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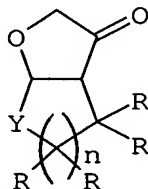


wherein X and Y, independently, are selected from the group consisting of O, S, and NR; each R, independently, is selected from the group consisting of hydro, C<sub>1-4</sub>alkyl, aryl, C<sub>1-3</sub>alkoxy, and C<sub>1-2</sub>alkylenearyl; and n is 1, 2, or 3, to provide a substituted furan-2-one;

(c) reducing the substituted furan-2-one of step (b) to provide a reduced product, then hydrolyzing the reduced product to provide a product having a structure

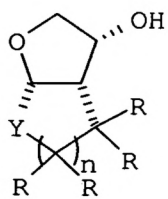


(d) oxidizing the product of step (c) to provide a product having a structure

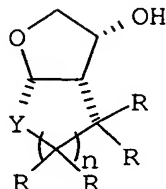


; and

(e) reducing the product of step (d) to provide the compound having a structure



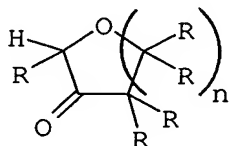
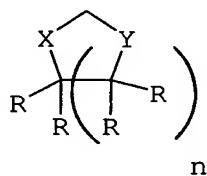
8. A method of preparing a compound having a structure



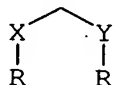
comprising:

(a) optionally reacting (5S)-hydroxymethyl-5H-furan-2-one with a compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one to provide a protected furan-2-one;

(b) subjecting (5S)-hydroxymethyl-5H-furan-2-one or the protected furan-2-one of optional step (a) to a photochemical reaction in the presence of a compound having a structural formula



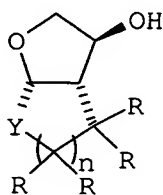
, or



wherein X and Y, independently, are selected from the group consisting of O, S, and NR; each

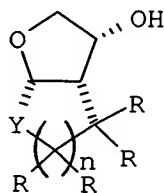
R, independently, is selected from the group consisting of hydro, C<sub>1-4</sub>alkyl, aryl, C<sub>1-3</sub>alkoxy, and C<sub>1-2</sub>alkylenearyl; and n is 1, 2, or 3, to provide a substituted furan-2-one;

(c) reducing the substituted furan-2-one of step (b) to provide a reduced product, then hydrolyzing the reduced product to provide a product having a structure



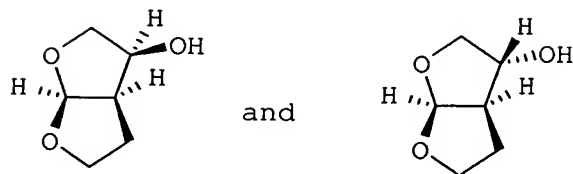
(d) subjecting the product of step (c) to a Mitsunobu inversion utilizing triphenylphosphine, *para*-nitrobenzoic acid, and diisopropylazodicarboxylate; and

(e) saponifying the product of step (d) to provide the compound having a structure





9. A method of preparing

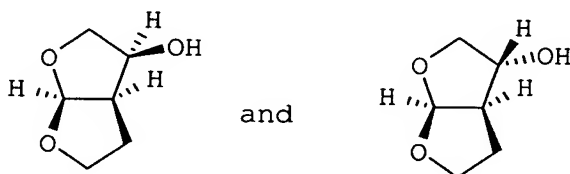


comprising:

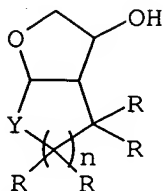
(a) optionally reacting (5R)-hydroxymethyl-5H-furan-2-one with a compound capable of positioning a protecting group at the hydroxy position of (5R)-hydroxymethyl-5H-furan-2-one to provide a protected furan-2-one;

(b) subjecting (5R)-hydroxymethyl-5H-furan-2-one or the protected furan-2-one of step (a) to a photochemical reaction in the presence of 1,3-dioxolane to provide a 1,3-dioxolan-substituted furan-2-one; and

(c) reducing the 1,3-dioxolan-substituted furan-2-one of step (b) to provide a reduced product, then hydrolyzing the reduced product to provide products having the structures



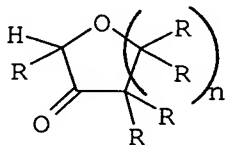
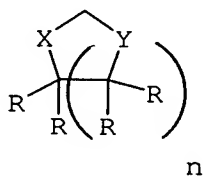
10. A method of preparing a compound having a structure



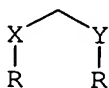
comprising:

(a) optionally reacting a hydroxymethyl-5H-furan-2-one with a compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one to provide a protected furan-2-one;

(b) subjecting the hydroxymethyl-5H-furan-2-one or the protected furan-2-one of step (a) to a photochemical reaction in the presence of a compound having a structure

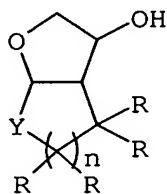


, or



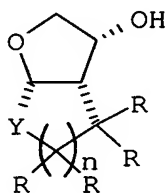
wherein X and Y, independently, are selected from the group consisting of O, S, and NR; each R, independently, is selected from the group consisting of hydro, C<sub>1-4</sub>alkyl, aryl, C<sub>1-3</sub>alkoxy, and C<sub>1-2</sub>alkylenearyl; and n is 1, 2, or 3, to provide a substituted furan-2-one;

(c) reducing the substituted furan-2-one of step (b) to provide a reduced product, then hydrolyzing the reduced product to provide a product having a structure



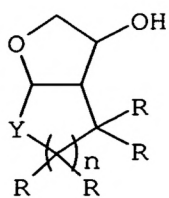
;

(d) oxidizing the product of step (c) to provide a product having a structure



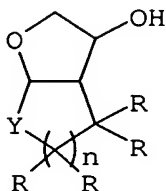
; and

(e) reducing the product of step (d) to provide the compound having a structure



11. A method of claim 10 wherein X and Y are O; each R is hydro; and n is 1 or 2.

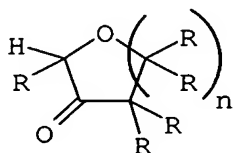
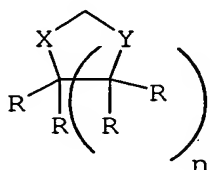
12. A method of preparing a compound having a structure



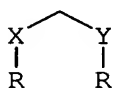
comprising:

(a) optionally reacting a hydroxymethyl-5H-furan-2-one with a compound capable of positioning a protecting group at the hydroxy position of (5S)-hydroxymethyl-5H-furan-2-one to provide a protected furan-2-one;

(b) subjecting the hydroxymethyl-5H-furan-2-one or the protected furan-2-one of step (a) to a photochemical reaction in the presence of a compound having a structure

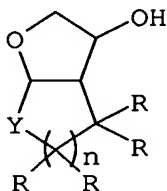


, or



wherein X and Y, independently, are selected from the group consisting of O, S, and NR; each R, independently, is selected from the group consisting of hydro, C<sub>1-4</sub>alkyl, aryl, C<sub>1-3</sub>alkoxy, and C<sub>1-2</sub>alkylenearyl; and n is 1, 2, or 3, to provide a substituted furan-2-one;

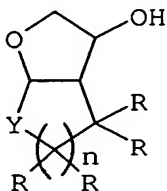
(c) reducing the substituted furan-2-one of step (b) to provide a reduced product, then hydrolyzing the reduced product to provide a product having a structure



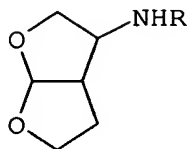
;

(d) subjecting the product of step (c) to a Mitsunobu inversion utilizing triphenylphosphine, *para*-nitrobenzoic acid, and diisopropylazodicarboxylate; and

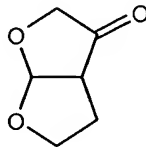
(e) saponifying the product of step (d) to provide the compound having a structure



13. A method of preparing a compound having a structure



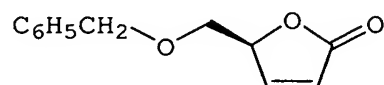
wherein R is selected from the group consisting of hydro, C<sub>1-4</sub>alkyl, aryl, C<sub>1-3</sub>alkoxy, and C<sub>1-2</sub>alkylenearyl, comprising subjecting a compound having a structure



to a reductive amination using an amine having a structure RNH<sub>2</sub>.



14. A method of preparing (5S)-5-benzyl-oxymethyl)-5H-furan-2-one having a structure



comprising the steps of

(a) subjecting (±)-1-(benzyloxy)-but-3-en-2-ol to an enzymatic acylation using immobilized lipase PS-30 and isopropenyl acetate to provide (S)-1-(benzyloxy)-but-3-en-2-ol;

(b) reacting the product of step (a) with acryoyl chloride to provide (S)-1-(benzyloxy)-but-3-en-2-yl acrylate; and

(c) interacting the product of step (b) with Grubbs catalyst  $(Cl_2(PCy_3)(IMes)Ru=CHC_6H_5)$  to provide (5S)-5-(benzyloxymethyl)-5H-furan-2-one.